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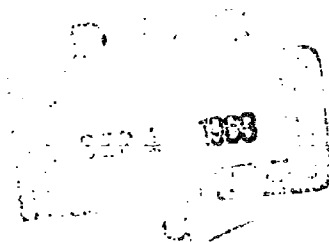
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16

GAS CHROMATOGRAPHY OF REACTIVE INORGANIC GASES

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Japan Analyst, Vol. 12, 1963, pp 450-457

A complete glass apparatus was constructed for the purpose of devising a gas chromatographic method for reactive materials. The efficiency of a column employing tetrafluoroethylene powder as the carrier and trifluorochloroethylene oil as the stationary liquid phase was investigated and chromatograms were obtained of common reactive gases such as oxides of nitrogen, halogens, and hydrogen halides.

The optimum conditions were as follows: Daifloil No. 3/Polyfluoron powder 30-60 mesh; 50/100 wt. ratio; column diameter 6 mm; room temperature. These conditions were sufficient to withstand a wide variety of materials.

1. INTRODUCTION

Heretofore, investigations in gas chromatography of highly reactive compounds have been in the development of non-corroding carriers and stationary liquid phases, especially with the use of chromatographic columns using fluorinated resins. There also have been various reports¹⁻³⁾ on the analysis of halogens and halogenated materials. The present authors, in connection with the analysis of factory gases and components of air pollutants, have made gas chromatographic determinations on samples containing highly reactive gases and have discussed the equipment, separation columns, and methods involved. They have discussed the following: (1) the general conditions for the use of fluorinated resins on the market; (2) the suitability for use with NO_2^* ; (3) a supplementary test, including the actual problems of quantity and operating

* Although there are reports of separation by means of silica gel⁴⁾, silicone grease⁵⁾, or by using reaction tubes⁶⁾, these were not used in this study.

conditions, for chlorine and bromine; (4) adaptability of the method to compounds such as HCN, HCl, and NOCl where details are unclear in the literature. The results were intended to widen the usable limits of the various reactive gases as well as to determine methods which would stand up sufficiently under actual use and to recognize the problems arising in their use. These experimental results are reported herein.

2. EQUIPMENT

The gas chromatograph flow system was constructed entirely of hard glass; all of the equipment other than the recorder and power source was home-made. To the 6 mm I.D. glass tube sample port, a similar diameter glass tubing approximately 1 cm long was attached at right angles. This was covered with silicon rubber. The entrance port was connected to the chromatographic column with a glass tubing 20 cm long, 4 mm I.D. and wrapped with glass tape to maintain the temperature. This could also be heated with a nichrome wire when necessary. The column was a unit constructed from glass tubing 6 mm I.D. and 1.5 m in length. Three meters, 6 m and 12 m lengths were also used by turning the tubing back and forth on itself. The joint of the column was connected with an approximately 2-cm length of trifluorochloroethylene (Daifluoron) pipe. In order to prevent leaks, the joint was sealed with epoxy (Supercemedyne) resin. In this manner, there was no leakage of carrier gas even with a 12-meter column. In changing the columns, the seam was lightly flamed and could easily be taken apart.

A glass enclosed platinum wire (10 μ) was used as a detector. The reference and detector cells were aligned and inserted in an aluminum block of approximately 8 cm³ dimensions, which was wrapped with glass tape to maintain the temperature. A cell current of 250 mA was used. The chromatographic column was bundled and placed in an approximately 6 cm thermostated tubing. The outside was wrapped with nichrome wire and the temperature was regulated from room temperature to 150° C. Furthermore, when cooling was necessary, only the column was placed in a water vessel and cooled with ice. A schematic diagram of the equipment is illustrated in Fig. 1.

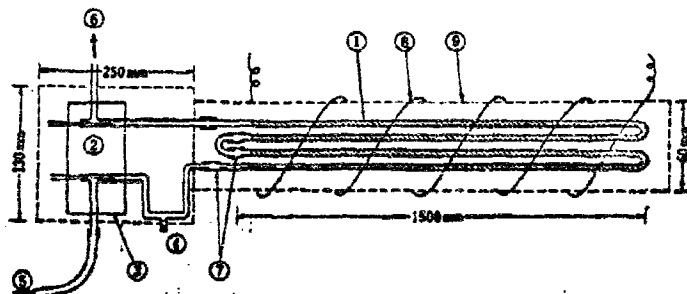


Fig. 1 Diagram of apparatus

- ① Column ② Pt-katharometer ③ Al block ④ Sample inlet ⑤ He inlet
 ⑥ He exit ⑦ Daisson conjunction ⑧ Nirom wire ⑨ Thermometer

The carrier gas was chiefly He. With the samples used in this research, no difference was obtained between H_2 or He. The samples were introduced with a hypodermic syringe. The sliding part was used with or without trifluorochloro ethylene oil. The needle was made either of stainless steel or a drawn-out pointed glass tubing. The needle-to-tubing joint was sealed on the outside with epoxy resin. When not in use, the syringe was kept in a dessicator filled with silica gel.

3. SAMPLES

It was extremely difficult to obtain pure gas samples. However, too much emphasis on the preparation of samples would not have been suitable for a discussion of chromatographic columns; therefore, several impure materials, to the extent that they did not interfere with the determination of the operation or system, were used in the following manner:

NO . In a nitrogen atmosphere, sodium nitrite was reduced with KI and dilute H_2SO_4 and stored in a glass container.

NO_2 . Nitrogen dioxide was obtained from the thermal decomposition of lead nitrate. It was passed through a P_2O_5 tube and trapped in a container cooled with dry ice. The air was removed and the trap was brought back to room temperature. For the slide-fitting, trifluorochloro ethylene grease was used. Gas mixtures from the NO obtained from pouring concentrated H_2SO_4 [sic] on copper was also used.

Cl_2 . From bombs and stored in glass container with silica gel.

Br_2 . From the saturated vapor above liquid bromine.

HCl . Concentrated H_2SO_4 dripped into concentrated HCl .

HBr . Concentrated H_2SO_4 dripped onto KBr .

NOCl. Chlorine and NO mixed in equivalent amounts and left standing.

HCN. Concentrated H_2SO_4 dripped onto KCN.

NH_3 . Concentrated NaOH solution dripped into concentrated ammonia water and dried by passing through a magnesium perchlorate tube.

N_2O . Gas from a bomb and stored in a glass container.

CO_2 . Gas from a bomb and stored in a glass container.

SO_2 . Concentrated H_2SO_4 dripped onto Na_2SO_3 .

O_3 . Approximately 1% concentration of ozone from a generator and used directly.

4. COLUMN MATERIAL

The fluorinated resin, tetrafluoroethylene powder (Polyfluoron) which is used as raw material in plastics, was manufactured by the Osaka Kinzoku Co. and trifluorochloroethylene powder (Daifluoron) were used as carriers. Since these materials are easily compactible soft powders and differ in form from the usual carriers, even carefully heated and sintered materials were tried. Moreover, although the material (Simalite) flame fused at the Shimazu Manufacturing Co. was used, it was difficult to absorb more than 1% of the fixed liquid phase to the flame-granulated material. Thus, this was unsuitable for the present research and was therefore not used. Although the aforementioned material could not be separated to the exact mesh size, it was first separated into 30 - 60 mesh sizes and used after eliminating the clumped scraps and the extremely minute sizes. Approximately half of the materials on the market were usable. Although there is entirely no difference in shape between tetrafluoroethylene and trifluorochloroethylene powders, the latter has a slight elasticity and in filling the columns there was a feeling of lack of uniformity. Furthermore, it was established that when soaked with trifluorochloroethylene oil, after a long period of use, there is a tendency of the usable time to become poor and the separating capacity of the column to become inefficient. One reason for this is thought to be that the carrier becomes saturated with the fixed liquid phase. For this reason, the former was utilized.

Trifluorochloroethylene oils (Daifloil) Nos. 1, 3 and 50, manufactured by the Osaka Kinzoku Co., were investigated as the fixed liquid phase. Although these have characteristics which overlap, there are still some variations. Near room temperature, the volatility of No. 1 is strong; No. 50 is greasy, and No. 3 was suitable for the aforementioned samples. No. 3 was fractionated at 0.1 mm Hg pressure from 120-150° C. The usable temperature conditions where these oils for the liquid phase are stable are presented in Table 1.

Table I Allowable operation temperature of liquid phases

Liquid phase	Evaporative loss (mg)	Allowable temp. limit (°C)
Daifloil No. 1	300	50
Dimethylsulfolane	127	80
Daifloil No. 2	93	60
<i>s,s'</i> -Oxydipropionitrile	76	70
Daifloil No. 50	7	120
Tricresylphosphate	2	150

The volatilities of these oils are compared with other liquid phases whose usable limits are known.

Approximately two ml of each liquid was taken in weighing bottles of 2.5 cm mouth and 3 cm depth and thermally vaporized under the same conditions in a thermostat at 92° C for three hours. According to these results, Daifloil No. 3 indicated almost the same characteristics as dimethyl sulfolane, and can be considered the best for use at room temperature.

With respect to the carrier, the liquid phase could uniformly coat up to 30 wt % (i.e., liquid/carrier at 50/100 weight ratio). Experimentally, the best was 30%.

When the diameter of the column was changed, partial coagulation occurred with 4 mm I.D. and the flow of the carrier gas became difficult. Thus, it was necessary to have chromatographic columns slightly larger than normal. With 6 mm I.D. these inconveniences did not arise and a length up to 12 m was used without any special differences when compared to ordinary gas chromatography. Furthermore, it could be used for a long period of time.

Next, the degree of polarity, a characteristic of the liquid phase, was determined. Following Rohrschneider's suggestion⁽¹⁾, the result of the determination of the relative positions of the peaks of n-butane and 1,3 butadiene were as follows: With a column length of 12 m and at 19° C, butane appeared at 16.7 cm and 1,3 butadiene at 17.5 cm (chart speed 1 cm/min) when measured from air and the polarity was log 1.05 0.02. According to this, the filler belongs to the group whose polarity is relatively small, resembling the unsaturated hydrocarbons (for example, liquid paraffin, triisobutylene). The chromatograms of hydrocarbons are illustrated in Fig. 2.

Furthermore, with respect to the separation, when the theoretical plates were determined using n-butane, it was found to be inferior to ordinary separation columns. The experimental values are compared in Table II.

This inferiority can be attributed to such things as non-uniformity of the carrier and the column diameters.

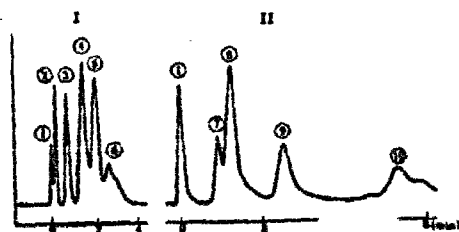


Fig. 2 Gas chromatogram of hydrocarbons

- I—Column: Daifloil No. 3 15% / Chromosorb.
6m; Temp.: 30°C; Carrier gas: H₂, 40 ml/min
- II—Column: Daifloil No. 3 15% / Chromosorb.
3m; Temp.: 130°C; Carrier gas: H₂, 50 ml/min
- ① Air ② Ethane + Ethylene
③ Propan + propylene ④ i-Butane
⑤ n-Butane ⑥ Butene ⑦ Cyclohexane
⑧ Benzene + n-Heptane ⑨ Toluene
⑩ Xylenes

Table II Theoretical plate number of n-butane

Column	T. P. N.
Daifloil No. 3/Chromosorb	1100
Daifloil No. 3/Polyflon	570
n-Hexadecane/Chromosorb	1400
n-Hexadecane/Polyflon	600

Liquid 20 wt%. 6m. Temp. 30°C, Carrier gas He 50 ml/min

Improving the separation efficiency under the conditions of its use was difficult to achieve. Besides this, when tetrafluoroethylene powder used as carrier absorbed propane at low temperatures and expelled it at room temperature, when compared to C-22, its desorption is delayed and even at room temperature there is some absorption.

5. EXPERIMENTAL RESULTS

The experimental results for separation of all samples in a column using Daifloil No. 3 and Polyflon powder in a 50/100 wt ratio are presented below.

The Nitrogen Oxides

NO. Used together with air or nitrogen. There was slight tailing of the NO peak when compared to the air peak. At lower temperatures (10°C) it was found on many occasions that the apex of the peak divided into two and thus the separation is possible. However, with columns up to 12 m the

separation was not definite.

N_2O . A sharp peak after the CO_2 peak was obtained. Both were completely separated and each could be satisfactorily determined quantitatively. In general, vapor-liquid gas chromatography, the separation of N_2O and CO_2 is difficult and in quantitative analysis, silica gel is used. Thus this characteristic of fluorinated resins is thought to be an advantage.

NO_2 . Nitrogen dioxide appears after N_2O ; however, it displays an unusual front-tailing peak. This peak begins to emerge in a section of the previous N_2O . Even with Daifloil No. 1 there was no difference in the appearance. With increasing temperature of the column, the peak sharpened and when increased to $50^\circ C$, its area could be measured fairly accurately. The tendency of the peak to improve following the change of temperature can be thought to indicate a lower column temperature with respect to the boiling point of NO_2 . However, raising the temperature is undesirable for separating other components such as N_2O . Furthermore, the characteristic of the NO_2 peak is such that according to the conditions, unusual changes in shape occur. This unusual tendency arises as follows: (1) when the sample is large; (2) when analysis is made after other samples have been passed through. In these situations it was determined that the effect of temperature change was great. It is thought that the residues of other components in the column were the cause. The possibility of quantitative analysis resulted from re-cycling the NO_2 sample (air, NO and N_2O gaseous mixtures). The chromatograms experimentally obtained are illustrated in Fig. 3 and 4.

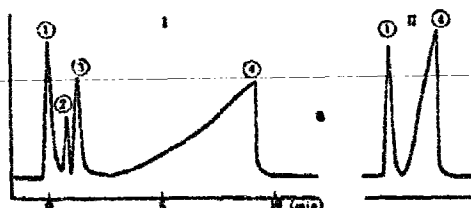


Fig. 3 Gas chromatogram of oxides of nitrogen and carbon dioxide

I: $15^\circ C$ II: $55^\circ C$

① Air+ NO ② CO_2 ③ N_2O ④ NO_2

Carrier gas: He 50ml/min

In the analysis of NO_2 it was necessary to carefully store and insert the NO_2 sample. In storage, the solution was to use Daifloil No. 50 grease on the stopcock of the flask. However, storage of dilute samples over long periods was difficult. The sample collection occurred after passing

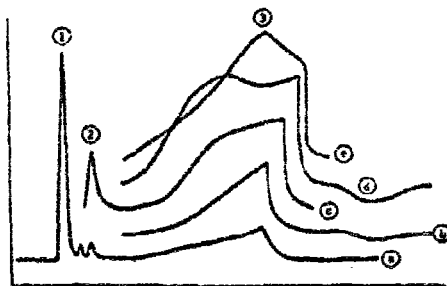


Fig. 4 Distortion of nitrogen dioxide peak

①-⑤ Showing different sample amount 0.1-
2ml ① Air ② N_2O ③ NO_2

through the silicone rubber, but it was necessary to isolate this section, other than during collecting, from the main part of the NO_2 filled flask by means of the stopcock. Unless this was done the silicone rubber became tacky after two or three days and could not be used. In the experiment to determine NO_2 quantitatively, it was found that the cause of the re-appearance of the peak was the syringe itself. Without grease and using a glass needle, it was possible to obtain relatively good results. The results are illustrated in Fig. 5.

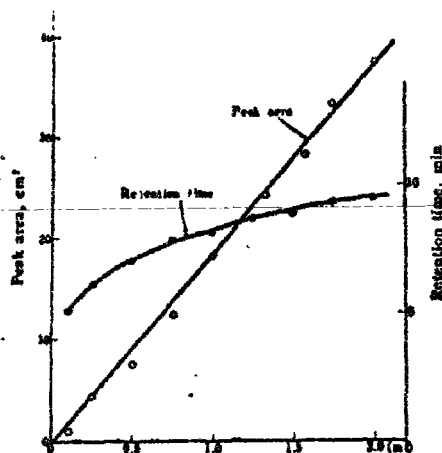


Fig. 5 Calibration curve of NO_2

Temp.: 23°C Carrier gas: He. 50ml/min

Moreover, it was determined that there occurred a shift in the position of the peak depending upon a change in sample

size. This is also illustrated in Fig. 4. A similar tendency was observed when the sample or column contained moisture. The effect of temperature on the shift of the peak is even more unusual than that caused by other components. For example, at lower temperatures it emerges after the chlorine peak, but at higher temperatures it appears before this peak while the largest distortion occurred at 50° C.

The relative retention times of all samples, the theoretical plate number, and relative sensitivities are given in Table IV. (The results obtained for the following samples are also presented.)

Table IV Experimental data on some reactive gases

Compound	Relative retention time (air=1)		Theoretical plate number (air=1500) (20°C)	Relative peak area carrier gas He (air=1)
	(20°C)	(70°C)		
NO	1.00			
HCl	1.257	1.15	1230	1.04
CO ₂	1.27		1250	1.52
N ₂ O	1.40		1300	1.25
H ₂ S	1.37		1200	1.12
HBr	1.50†	1.30	1300	
HCN	1.66		1090	1.86
SO ₂	2.22		770	1.67
Cl ₂	2.56	1.82	880	1.75
NOCl	3.12		830	
NO ₂	3.78†	1.60	430†	1.74†
Br ₂		5.00	660††	†
NH ₃	1.30†		†	†
Ethane	1.25			
Propane	2.05		860	
n-Butane	4.49		570	

† Varying with water, sample amount etc. †† 70°C

Halogens. Although the application of fluorinated resins to chlorine and bromine has been reported previously, from the supplementary experiments and even from our column, good separation and sharp peaks were obtained. Thus the suitability to the analysis of these samples was determined. The results of quantitative analyses obtained from varying the amount of the same sample are shown in Table III.

Table III Quantitativity of Cl₂ analysis

Sample amount(ml)	Cl ₂ peak area(mm ²)	Cl ₂ (vol%)
1.0	1368	21.03
0.8	1111.5	21.55
0.6	807.5	21.47
0.4	541.5	22.53
0.2	285	22.91
0.1	142.5	21.99

The column was 6 m and the temperature was 15° C. The percentage of chlorine was obtained from the peak areas. When using the hypodermic syringe a certain amount of error was unavoidable. From these results, it is thought that there is sufficient quantitative character for actual use.

It was found that on the repeated introduction of a fixed quantity of the sample, the peak areas became larger. With chlorine, an approach to a fixed value was seen. This is illustrated in Fig. 6.

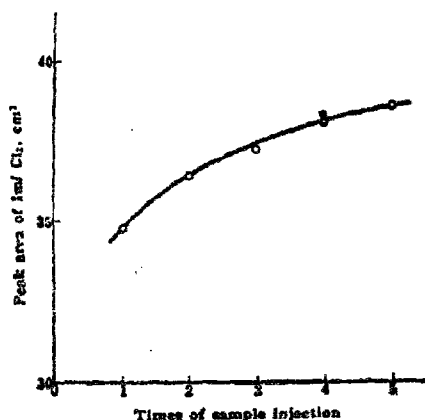


Fig. 6 Variation of Cl₂ peak area

Temp.: 20°C Carrier gas: He, 40 ml/min

With bromine, this tendency again was unusual. It was found that on repeated sample introduction and when a slight pause was made before introduction, the peak areas differed. For example, with the same sample, at an injection time of eight minutes the area was 12.7 cm² and at 20 minutes, 9.83 cm². In quantitative determinations there is a necessity to consider this tendency from the standpoint of handling standard samples and the effect of mixture components. Again, especially in cases where the analysis of bromine is the primary purpose, it is desirable to use higher temperatures with highly overlapping liquid phases.

NOCl. A symmetrical peak following chlorine was obtained for NOCl. Moreover, when successive determinations of nitrogen oxides and chlorine are made, this peak appears and the chromatogram changes.

Hydrogen halides. Both HCl and HBr emerge before Cl₂ and Br₂. HCl appear simultaneously with C₂ hydrocarbon and CO₂. These peaks exhibited some tailing and in the presence

of much moisture the tailing became severe making the analysis of small samples difficult. Furthermore, in this situation the retention volume becomes large and the change in retention volume with changes in sample size is unusual. These results are shown in Fig. 7.

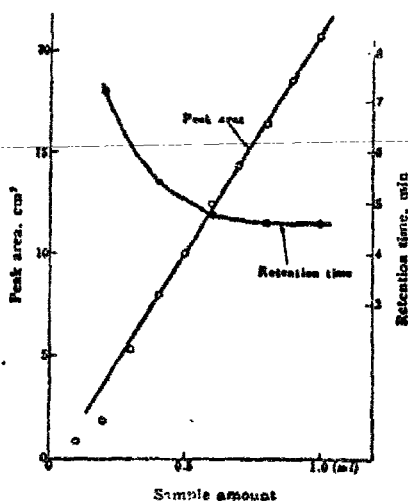


Fig. 7 Calibration curve of HCl

Temp.: 15°C Carrier gas: He, 46 ml/min

At 15° C in the presence of moisture, a chromatogram for complete separation of CO₂ was obtained. The above chromatogram is shown in Fig. 8.

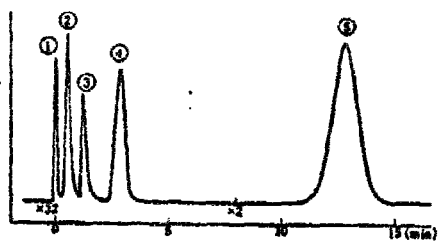


Fig. 8 Gas chromatogram of HCl, HBr, Cl₂ and Br₂

Column: 6 m Temp.: 70°C
Carrier gas: He, 46 ml/min
① Air
② HCl ③ HBr ④ Cl₂ ⑤ Br₂

HCN. It was found that the HCN peak was quite symmetrical and its quantitative analysis was satisfactory. The

position of the peak was between $\text{HBr} \sqrt{\text{sic}}$ and SO_2 . However, the position of the peak with regard to $\text{HBr} \sqrt{\text{sic}}$ was reversed on occasions.

Fig. 9 is a typical chromatogram for the sample above.

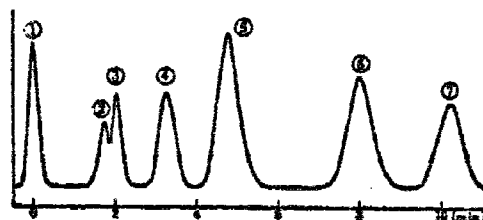


Fig. 9 Typical gas chromatogram of tested gases

Column: 6m Temp: 20°C
Carrier gas: He, 40 ml/min ① Air ② CO_2
③ H_2S ④ HCN ⑤ SO_2 ⑥ Cl_2
⑦ NOCl

NH_3 . Ammonia displays an unusual tailing. The effect of moisture was found to be even greater than in HCl and the determination of small samples was difficult. Furthermore, analysis could not be made when the order was reversed with acid samples and, in this case, it was necessary to inject the samples repeatedly.

The calibration curve is illustrated in Fig. 10 and a chromatogram in Fig. 11.

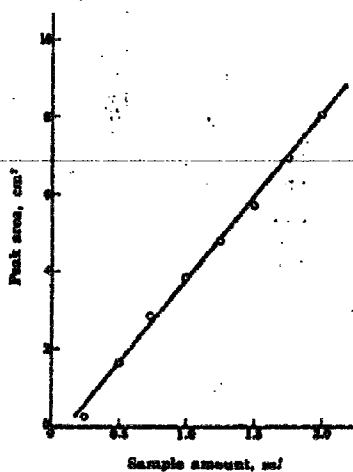


Fig. 10 Calibration curve of NH_3
Temp: 40°C Carrier gas: H_2 , 60 ml/min

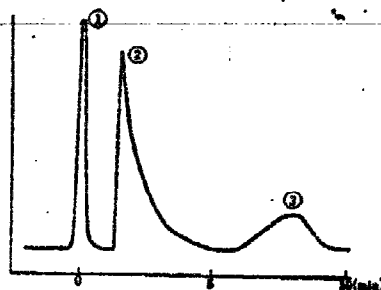


Fig. 11 Gas chromatogram of NH_3
Temp: 40°C Carrier gas: H_2 , 60 ml/min
① Air ② NH_3 ③ Water

PRELIMINARY RESULTS OF SEED SELECTION IN THE QUANG NINH AREA

- North Vietnam -

[Following is a translation of an article by
Mai Van Dong, Bureau of Agriculture, Quang
Ninh, in the Vietnamese-language periodical
Khoa Hoc Ky Thuat Nong Nghiep (Agricultural
Sciences & Technology), Hanoi, October 1964,
pp 592-594.]

Many species of rice can be found in the northern
sector of Quang Ninh. Excellent species are developing rap-
idly but many bad species still exist. Many species have
been cultivated for many years but due to lack of care and
selection some of them are undergoing mixing and regression.

Investigation in some cooperatives (at Dam Ha) has
shown the large extent of mixing in some rice species (Table I)

Table I
Investigation at Dam Ha

① Hợp tác xã	② Giống lúa	Tỷ lệ lẫn ③ (%)
Bắc-sơn	④ Chiêm trắng	20
	⑤ Lúa mùa	
	Ngân-kêng-hồng	16
	Chín-chí-chợa	13
Yên-dịnh	Chiêm trắng	15
Yên-hàn	Chiêm trắng	12
	⑥ Dục-tái-chim (lúa mùa)	15

[Legend]: 1) Cooperative; 2) Rice species;
3) percent component; 4) white "chiem"; 5)
Main Crop; 6) Dop Tai Chim (main crop).

The increasingly poor seeds have given unstable yields
(Table 2).

Table 2
Rice Yields (piculs/ha) at Tien Yen

① Tên xã	1959	1960	1961	1962
Dông-ngô	20	19	12	14
Hải-lạng-dông	24	22	18	19
Yên-thân	18	10	15	15

[Legend]: 1) Villages.

Thus, it is important to select excellent seeds that
will give high and stable yields.

I. COMPARISON OF SEEDS

Rice yields have been observed through a number of
crops at the Ha Coi and Doan Tinh [work] camps and some co-
operatives. Results are, as follows:

1. Spring "chiem" rice. Comparison of yields for
spring crops over several years (sown 19 March, planted 17
April) indicate that "Nam Ninh" rice has been the most prom-
ising species, that its yield is almost stable and is higher
than that of white "chiem" (Pac Pha). (Table 3)

The duration of growth of "Nam Ninh" is five to seven
days shorter than that of white "chiem" which is an important
factor in labor scheduling. "Nam Ninh" plants are tougher
and develop faster than white "chiem". The "Nong Nghiep" I
seeds of "Nam Ninh" give strong stems, big ears, numerous
grains but are susceptible to "dao on" and "tiem lua" diseases.
Further study in this area is needed.

According to current data, "Nam Ninh" rice should be
readily adaptable to local conditions. It is being cultivated
by many cooperatives and occupies 70-80 per cent of the area
for "chiem" varieties.

Table 3

① Tên giống	② Thời gian sinh trưởng (số ngày)	③ Năng suất (tạ/ha)		
		1961	1962	1963
Nam-ninh	110-115	20	27,3	21,25
Nông nghiệp I	103	10,5	27,0	21,25
Chiêm âm	115-120	19,0	28,0	6,87*

[Legend]: 1) Species; 2) Duration of Growth (Days); 3) Yield (Piculs/ha).

* In 1963, blossoms fell prematurely

2. Main-crop rice. In addition to species existing locally, some species have been imported from China and from the delta regions in recent years. These species are tentatively classified into three groups:

- Local species: "loong xay chien," "tan thuy lien," "dop tai chim," "dai hong coc."

- Species brought from the delta region: "quyet tam" 813, "di huong," "tam thom."

- Species imported from Dong Hung (China): "tan tay duong," "chuc tuy," "moc toan," "chung thu," "ke nam ai," "thap thach kiem," "dong thu bo," unknown No. 1, unknown No. 2.

Yields are summarized in Table 4.

Investigations have led to the following conclusions:

- Among local species, "loong xay chim" and "tan thuy tien" gave the highest and most consistent yields. "Loong xay chim" is tasty, and it has been cultivated in this area for a long time. However, it has become mixed and needs to be selected. "Tan thuy lien" seeds produce tall and strong plants with large grains. The unhulled grains are red, and they are low quality grains. This species, however, can withstand alkalinity and resist diseases.

- Of the species imported from the delta, "di huong" produces uniform ears. Its short plants are not likely to collapse. "Tam thom" and "quyet tam" 813 are not adaptable.

- In the imported group, "tan tay duong" has been cultivated for a relatively long time. It is readily adaptable to local conditions, and it is becoming increasingly popular. A shortcoming of the "tan tay duong" is its tendency to collapse and to shed its grains. Selection is needed. At present, "tan tay duong" represents a large portion of rice grown in rice-rich huyen such as Dam Ha, Ha Coi and Mong Cai.

Table 4

① Tên giống	② Thời gian sinh trưởng (số ngày)	③ Năng suất (tạ/ha)		
		1961	1962	1963
Công-xây-chim	135-140	21	36,00	23
Tân-thủy-liên	150	39,3	39,20	22,8
Đẹp-tài-chim	135	20,0		
Đại-hồng-oắc			21,70	
Quyết-tâm 813	165-170		34,0	20,5*
Di hương	150		30,0	17,0
Tâm thơm	160		26,0	
Tân-tây-duong	152		30,0	26,8
Mộc-toàn	155		44,0	27,0
Chúc-tuy	155		37,0	7,5
Chứng-thu	157		40,0	27,0
Đông-thu-hồ	159			18,6
④ Vô danh số 2	159			26,43

[Legend]: 1) Species; 2) Duration of Growth (days); 3) Yield (piculs/ha); 4) Unknown No. 2.

* In 1963, the ears came late due to diseases

II. SEED SELECTION

In 1963, various [work] camps and cooperatives carried out seed selection on the basis of observations made by the seed union. Seeds were selected from fields which had received extensive care and high yield (Table 5).

Table 5

Data from Some Cooperatives in Dam Ha on Fields
Chosen from Seed Selection

① Công việc	② Hợp tác xã Bắc-sơn		③ Hợp tác xã Yên-dinh	
	④ Ruộng giống	⑤ Ruộng sản xuất	Ruộng giống	Ruộng sản xuất
⑥ Cây	3 lượt	2 lượt	3 lượt	2 lượt
⑦ Bón	3 lượt	2 lượt	3 lượt	2 lượt
⑧ Bón lót/1 la	20 gánh phân chuồng + 40 gánh đất + 40 kg P	10 gánh phân chuồng + 15 gánh đất + 15 kg P/1 la	15 gánh phân chuồng + 10 gánh đất + 20kg P, vôi + 10kg N	6 gánh phân chuồng + 35 gánh đất + 3 kg xác mắm
⑨ Bón thúc/1 la	33 kgN + 3kgP + 3 gánh phân chuồng	3 gánh phân chuồng	6 gánh phân chuồng + 2,5kg N	17 kg N
⑩ Làm cỏ	2 lượt	1 lượt	1 lượt	1 lượt
⑪ Năng suất (tạ/ha)	35	22	25	21

[Legend]: 1) Operation; 2) Bac Son Cooperative; 3) Yen Dinh Cooperative; 4) Selection Field; 5) Production Field; 6) Plowing; 7) Harrowing; 8) First fertilization 1La; 9) Additional Fertilizing 1 La; 10) Weeding; 11) Yield (piculs/ha); 12) 20 shoulder-loads manure; 13) +40 shoulder-loads soil; 14) +40 kg. phosphate; 15) 3 kg Nitrate + 3 kg phosphate; 16) 20 kg phosphate lime + 10 kg nitrate; 17) 6 shoulder-loads manure; 2.5 kg nitrate; 18) + 3 kg fish residue; 19) 3 kg nitrate.

- The number of stalks per clump in the "Nam Ninh" species are given in Table 6 for seed selection fields and production fields.

Where single stalks were planted for the purpose of seed selection, the growth was more vigorous, the number of grains per ear was double its counterpart in production fields where multiple stalks were planted. Most cases of single stalk planting occurred during the main-crop season in 1963.

- For single stalks, the number of ears per clump, the number of grains per ear were larger than in the case of multiple-stalk planting for production. And the proportion of empty grains was significantly lower.

Efforts in seed selection from 1962 to 1963 in the northern sector of Quang Ninh provided many fields for single-stalk planting and large quantities of seeds for production during subsequent years in various cooperatives.

Table 6

① Đơn vị	② Số cành cây	Số nhánh đẻ ③ 1 khóm		Số hạt 1 bông	
		④ Nhiều nhất	⑤ Ít nhất	⑥ Nhiều nhất	⑦ Ít nhất
Yên-dinh	4-5	14	6	50	14
	1	12	2	108	60
Bắc-sơn	② nhiều.	13	4	50	22
	dánh 1	11	2	100	32

[Legend]: 1) Unit; 2) Number of stalks; 3) Number of shoots per clump; 4) Number of grains per ear; 5) maximum; 6) minimum; 7) Multiple stalks.

Table 7

①. Trung bình	② Chiêm trắng (Yên-dinh)		③ Chiêm trắng (Bắc-sơn)	
	④ Nhiều nhất	⑤ Ít nhất	⑥ Nhiều nhất	⑦ Ít nhất
⑧ Số bông 1 khóm	10	8	9	6,5
⑨ Số hạt 1 bông	27	82	38	75
⑩ Tỷ lệ hạt lép (%)	39	2,4	21,8	5,7

[Legend]: 1) Average; 2) White "chiem"; 3) multiple stalks; 4) single stalk; 5) ears per clump; 6) grains per ear; 7) hollow grains (%).

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